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# (54) FLAME-RETARDANT POLYPHENYLENE ETHER-BASED RESIN COMPOSITION

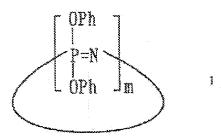
### (57) Abstract

PROBLEM TO BE SOLVED: To obtain the subject composition which excels in flame-retardancy, contains no halogen, has high melting point and low volatility, and does not lower its mechanical properties, molding processability, etc.. crosslinked including a specific resin and a phosphazene-based flame retardant.

SOLUTION: This composition is obtained including (A)100 pts.wt. of a resin comprising (i)1-99 pts.wt. of a polyphenylene ether resin and (ii) 99-1 pts.wt. of a polystyrene-based resin and

(B) 1-30 pts.wt. of crosslinked phosphazene-based flame retardant. The component B is preferably such a compound that a compound of formula I (m is 3-25; Ph is phenyl) and/or formula II [X1 is N=P(O)OPh or the like; Y1 is P(OPh)4 or the like; n is 3-10,000] is crosslinked with a crosslinking group such as o-phenylene, the crosslinking group interposes between two Os as a result of eliminating the phenyl group of the phosphazene compound, the content of the phenyl groups is 50-99.9% based on the total phenyl groups of the compound(s) of formula I and/or formula II, and there is no free hydroxyl group in the molecule.

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### CLAIMS -

# [Claim(s)]

[Claim 1] (A) The fire-resistant polyphenylene ether system resin composition object characterized by blending (B) bridge formation phosphazene system flame retarder 1 - 30 weight sections to the resin 100 weight section which consists of polyphenylene ether resin 1 - 99 weight sections and polystyrene system resin 99 - the 1 weight section.

[Claim 2] (A) The fire-resistant polyphenylene ether system resin composition object according to claim 1 as for which below 30 weight sections blended (C) triazine frame content compound to the resin 100 weight section of a component.

[Claim 3] (A) The fire-resistant polyphenylene ether system resin composition object according to claim 1 or 2 as for which below 50 weight sections blended the (D) inorganic filler to the resin 100 weight section of a component.

[Claim 4] (A) Claim 1 as for which below 30 weight sections blended the organic phosphorous compound which does not contain the (E) halogen to the resin 100 weight section of a component, a fire-resistant polyphenylene ether system resin composition object according to claim 2 or 3.

[Claim 5] (B) The bridge formation phosphazene system flame retarder of a component is a general formula (1).

In the inside m of [type, the integer of 3-25 is shown. Ph shows a phenyl group. The annular phenoxy phosphazene and the general formula (2) which are expressed with ]

$$X^{l} \xrightarrow{\text{OPh}} X^{l} \qquad (2)$$

The inside X1 of [type is a radical. - N=P(OPh) 3 or radical-N=P(O) OPh is shown, and Y1 is a radical. - P(OPh)4 or radical-P(O) (OPh)2 are shown. n shows the integer of 3-10000. Ph is the same as the above. At least one sort of phosphazene compounds chosen from the group which consists of straight chain-like phenoxy phosphazene expressed with ] are o-phenylene group, m-phenylene group, p-phenylene group, and a general formula (3). [Formula 3]

In the inside A of [type, -C(CH3)2-, -SO2-, -S-, or -O- is shown, a shows 0 or 1. It is the compound over which at least one sort of bridge formation radicals chosen from the group which consists of a bis-phenylene group expressed with ] come to construct a bridge. (a) This bridge formation radical intervenes between two oxygen atoms from which the phenyl group of the phosphazene compound was desorbed. (b) The content ratio of a phenyl group is 50 to 99.9% on the basis of the total of the above-mentioned phosphazene compound (1) and/or all the phenyl groups in (2). And Claim 1 which is the bridge formation phenoxy phosphazene compound which does not have a free hydroxyl group in (c) intramolecular, Claim 2, a fire-resistant polyphenylene ether system resin composition object according to claim 3 or 4.

[Claim 6] Claim 1, Claim 2, Claim 3, the fire-resistant polyphenylene ether system resin Plastic solid that can fabricate and obtain a fire-resistant polyphenylene ether system resin composition object according to claim 4 or 5.

### DETAILED DESCRIPTION

# [Detailed Description of the Invention] [0001]

[Field of the Invention] This invention relates to a fire-resistant polyphenylene ether system resin composition object and a fire-resistant polyphenylene ether system resin Plastic solid.

# [0002]

[Description of the Prior Art] Polyphenylene ether system resin is used for the application of autoparts, electrical machinery and an electronic item, OA equipment, Business Machinery Division, communication equipment, etc. from properties, such as the outstanding mechanical characteristic, an electrical property, and thermal resistance. For these applications, flameproofing of resin is needed from problems, such as exoergic ignition of internal components.

[0003] In order to give fire retardancy to polyphenylene ether system resin, the method of adding a flame retarder is common in front of resin shaping. As a flame retarder, an inorganic hydroxide, an organic halogenated compound, a halogen content organic phosphorous compound, red phosphorus, organophosphate, a phosphazene system

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### **CLAIMS**

## [Claim(s)]

[Claim 1] (A) The fire-resistant polyphenylene ether system resin composition object characterized by blending (B) bridge formation phosphazene system flame retarder 1 - 30 weight sections to the resin 100 weight section which consists of polyphenylene ether resin 1 - 99 weight sections and polystyrene system resin 99 - the 1 weight section.

[Claim 2] (A) The fire-resistant polyphenylene ether system resin composition object according to claim 1 as for which below 30 weight sections blended (C) triazine frame content compound to the resin 100 weight section of a component.

[Claim 3] (A) The fire-resistant polyphenylene ether system resin composition object according to claim 1 or 2 as for which below 50 weight sections blended the (D) inorganic filler to the resin 100 weight section of a component.

[Claim 4] (A) Claim 1 as for which below 30 weight sections blended the organic phosphorous compound which does not contain the (E) halogen to the resin 100 weight section of a component, a fire-resistant polyphenylene ether system resin composition object according to claim 2 or 3.

[Claim 5] (B) The bridge formation phosphazene system flame retarder of a component is a general formula (1).

[Formula 1]

In the inside m of [type, the integer of 3-25 is shown. Ph shows a phenyl group. The annular phenoxy phosphazene and the general formula (2) which are expressed with ] [Formula 2]

$$X^{1} + \begin{cases} OPh & \\ P=N & \\ \\ OPh & \\ \\ \end{pmatrix}_{R} Y^{1}$$
 (2)

The inside X1 of [type is a radical. - N=P(OPh) 3 or radical-N=P(O) OPh is shown, and Y1 is a radical. - P(OPh)4 or radical-P(O) (OPh)2 are shown. n shows the integer of 3-10000. Ph is the same as the above. At least one sort of phosphazene compounds chosen from the group which consists of straight chain-like phenoxy phosphazene expressed with ] are o-phenylene group, m-phenylene group, p-phenylene group, and a general formula (3). [Formula 3]

In the inside A of [type, -C(CH3)2-, -SO2-, -S-, or -O- is shown a shows 0 or 1. It is the compound over which at least one sort of bridge formation radicals chosen from the group which consists of a bis-phenylene group expressed with ] come to construct a bridge. (a) This bridge formation radical intervenes between two oxygen atoms from which the phenyl group of the phosphazene compound was desorbed. (b) The content ratio of a phenyl group is 50 to 99.9% on the basis of the total of the above-mentioned phosphazene compound (1) and/or all the phenyl groups in (2). And Claim 1 which is the bridge formation phenoxy phosphazene compound which does not have a free hydroxyl group in (c) intramolecular, Claim 2, a fire-resistant polyphenylene ether system resin composition object according to claim 3 or 4. [Claim 6] Claim 1, Claim 2, Claim 3, the fire-resistant polyphenylene ether system resin Plastic solid that can fabricate and obtain a fire-resistant polyphenylene ether system resin composition object according to claim 4 or 5.

## DETAILED DESCRIPTION

# [Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to a fire-resistant polyphenylene ether system resin composition object and a fire-resistant polyphenylene ether system resin Plastic solid. [0002]

[Description of the Prior Art] Polyphenylene ether system resin is used for the application of autoparts, electrical machinery and an electronic item, OA equipment, Business Machinery Division, communication equipment, etc. from properties, such as the outstanding mechanical characteristic, an electrical property, and thermal resistance. For these applications, flameproofing of resin is needed from problems, such as exoergic ignition of internal components.

[0003] In order to give fire retardancy to polyphenylene ether system resin, the method of adding a flame retarder is common in front of resin shaping. As a flame retarder, an inorganic hydroxide, an organic halogenated compound, a halogen content organic phosphorous compound, red phosphorus, organophosphate, a phosphazene system compound, etc. are known.

[0004] However, halogen content compounds, such as an organic halogenated compound and a halogen content organic phosphorous compound, pyrolyze at the time of resin shaping, although fire-resistant effectiveness is excellent, hydrogen halide is generated, and the corrosion of metal mold, degradation of resin, and coloring are caused. In addition, when resin burns by incineration, a fire, etc., it has harmful gas and smoke to living things, such as hydrogen halide, and the trouble which generates dioxin further and causes environmental pollution. Both halogen content compounds are using an antimony trioxide, and, generally the antimony trioxide is used as a fire-resistant assistant from fire retardancy improving remarkably. However, since an antimony trioxide has the misgiving of a carcinogenic compound, there is a problem in concomitant use with a halogen content compound. [0005] On the other hand, the flame retarders which do not contain a halogen are inorganic hydroxides, such as a magnesium hydroxide and an aluminum hydroxide, red phosphorus, organophosphate, a phosphazene system compound, etc. However, since fire retardancy is discovered with the water produced in a pyrolysis, fire-resistant effectiveness must add an inorganic hydroxide so much low therefore, but there is a fault that the property of resin original is spoiled, by carrying out abundant addition. Although fire-resistant effectiveness of red phosphorus is high, the resin composition object has the fault colored red. It is widely used from comparatively good fire-resistant effectiveness being acquired, and organophosphate is triphenyl phosphate (TPP) as a typical thing, The resorcinol screw (diphenyl phosphate) (RDPP) etc. is known (JP,S53-418,B, JP,S53-73248,A description).

however -- since these organophosphate is a liquid or a lower melting point solid-state, its volatility is high -- reducing the thermal resistance of resin greatly \*\*\*\* -- the blocking at the time of kneading -- oozing out (JUSHINGU) -- etc. -- there is a problem.
[0006]

[Problem(s) to be Solved by the Invention] In JP, H3-73590, B, the polyphenylene ether system resin composition object which blended the phosphazene system compound with polyphenylene ether system resin is indicated. In this gazette, while heat-resistant lowering of the constituent which uses polyphenylene ether, or this and polystyrene system resin as a resinous principle is effectively controlled by combination of a phosphazene system compound, it is indicated that the fire-resistant grant which was superior to phosphoric ester is attained. However, with a technique given in this gazette, polyphenylene ether and polystyrene system resin are set to the constituent used as a resinous principle. The fireresistant advanced fire retardancy which attains the fire-resistant assessment V-0 in the fireresistant examination as which the content of polystyrene system resin is specified to UL-94 [ excellent in the high resin composition object (for example, resin composition object whose content of polystyrene system resin is 30 to 90 weight %) cannot be given. [0007] Moreover, to JP,H7-216235,A, the flame-retardant-resin constituent which blended the salt of (1) phosphazene system compound and (2) cyanuric acid or isocyanuric acid, and a melamine compound with thermoplastics as a flame retarder is indicated. A resin composition object not generating volatile constituents, such as hydrogen halide, and having high fire retardancy, a mechanical characteristic, and hydrolysis-proof nature is indicated by this gazette. However, although this gazette has the concrete description which carried out flameproofing of the polyphenylene oxide resin (PPO, i.e., polyphenylene ether (PPE) resin) which is comparatively easy to carry out flameproofing There is no concrete description which carried out flameproofing of the denaturation PPE resin which blended the polystvrene system resin made very difficult [ flameproofing ]. When this invention person actually blends the above-mentioned flame retarder with high denaturation PPE resin (content of polystyrene system resin: 30 to 90 weight %) of the content of polystyrene system resin, It was impossible for dripping to have happened and to have given during combustion, sufficient fire retardancy (advanced fire retardancy which attains the fire-resistant assessment V-0 in the fire-resistant examination specified to UL-94) for this denaturation PPE resin. 100081

[Means for Solving the Problem] this invention person found out that sufficient fire retardancy for this resin could be given by blending a bridge formation phosphazene system flame retarder with the resin which consists of polyphenylene ether resin and polystyrene system resin, as a result of inquiring wholeheartedly that the above-mentioned technical problem should be solved. Moreover, this invention person found out that much more advanced fire retardancy could be given with this resin by blending a bridge formation phosphazene system flame retarder and a triazine frame content compound with the resin which consists of polyphenylene ether resin and polystyrene system resin.

[0009] As opposed to the resin 100 weight section which consists of (A) polyphenylene ether resin 1 - 99 weight sections and polystyrene system resin 99 - the 1 weight section according to this invention (B) The fire-resistant polyphenylene ether system resin composition object characterized by blending 1 - 30 weight section for a bridge formation phosphazene system flame retarder is offered.

[0010] Moreover, according to this invention, the resin 100 weight section which consists of (A) polyphenylene ether resin 1 - 99 weight sections and polystyrene system resin 99 - the 1 weight section is received. (B) The fire-resistant polyphenylene ether system resin composition object which blended (C) triazine frame content compound with the constituent which blended 1 - 30 weight section for the bridge formation phosphazene system flame retarder to the resin 100 weight section of the (A) component further below in 30 weight

sections is offered.

[0011] Moreover, according to this invention, the flame-retardant-resin constituent with which the organic phosphorous compound which does not contain below 50 weight sections and/or the (E) halogen for the (D) inorganic filler further was blended with the above-mentioned fire-resistant polyphenylene ether system resin composition object below as for 30 weight sections is offered.

[0012] Furthermore, according to this invention, the fire-resistant polyphenylene ether system resin Plastic solid which can fabricate and obtain various kinds of above-mentioned fire-

resistant polyphenylene ether system resin composition objects is offered.

[0013] [according to this invention] even if it is polyphenylene ether system resin with high content of inflammable high polystyrene system resin High-melting and the property of low volatility and resin [, such as a mechanical property and fabricating-operation nature, ] original are not reduced excluding a halogen, the blocking at the time of kneading — oozing out — etc. — it is satisfactory, and dripping cannot be caused during combustion, but it can be considered as the resin composition object equipped with the advanced fire retardancy which attains the fire-resistant assessment V-0 in the fire-resistant examination specified to UL-94.

[Embodiment of the Invention] [Resinous principle] Polyphenylene ether resin which is the (A) component of this invention is a following general formula, for example. [0015] [Formula 4]

[0016] Among [R1] [type, R2, R3, and R4 are the same or different, and show the alkyl group of a hydrogen atom or carbon numbers 1-3. It is the polymer which consists of a structural unit expressed with ]. The above-mentioned structural unit may be the homopolymer which consists of one sort, and polyphenylene ether resin of this invention may be a copolymer which consists of two or more sorts of the above-mentioned structural unit. [0017] As a concrete example of such polyphenylene ether resin For example, the Pori (2, 6-dimethyl 1, 4-phenylene) ether, the Pori (2, 6-diethyl 1, 4-phenylene) ether, The Pori (2-methyl 6-ethyl 1, 4-phenylene) ether, The Pori (2-methyl 6-propyl 1, 4-phenylene) ether, The Pori (2-ethyl 6-propyl 1, 4-phenylene) ether, The copolymer of 2 and 6-dimethylphenol and a 2, 3, and 6-trimethyl phenol, The copolymer of 2 and 6-diethyl phenol and 2, 3, and 6-trimethyl phenol, The copolymer of 2 and 6-diethyl phenol etc. is mentioned, and especially Pori (2, the 6-dimethyl 1, 4-phenylene ether) is desirable in it.

[0018] these polyphenylene ether resin - the 4th edition "experimental science lecture" - the 28th volume Polymer composition According to the laboratory procedure of a description, it

may be compounded [page / 182nd] easily.

[0019] Polystyrene system resin which is other one (A) component of this invention is a following general formula, for example. [0020]
[Formula 5]

shown. R6 shows the alkyl group of a halogen atom or carbon numbers 1-3.1 shows the integer of 0, or 1-3. It is resin which contains the structural unit expressed with ] at least 25weight % or more in the polymer.

[0022] As a concrete example of such polystyrene system resin, polystyrene, rubber denaturation polystyrene (high impact polystyrene), a styrene butadiene copolymer, a styrene butadiene acrylonitrile copolymer, etc. can be mentioned. High impact polystyrene is suitably used in it. The rubber denaturation polystyrene which denaturalized of rubber components, such as polybutadiene styrene copolymer rubber and EPDM, is included by polystyrene system resin of this invention.

[0023] Polyphenylene ether resin is [polystyrene system resin of the blending ratio of coal of polyphenylene ether and polystyrene system resin] usually 99 - 1 weight section to 1 - 99 weight section.

[0024] [Bridge formation phosphazene system flame retarder] As a bridge formation phosphazene system flame retarder which is the (B) component of this invention, it is a general formula (1), for example.

[0025]

[Formula 6]

[0026] In the inside m of [type, the integer of 3-25 is shown. Ph shows a phenyl group. The annular phenoxy phosphazene and the general formula (2) which are expressed with ] [0027]

[Formula 7]

[0028] The inside X1 of [type is a radical. - N=P(OPh) 3 or radical-N=P(O) OPh is shown, and Y1 is a radical. - P(OPh)4 or radical-P(O) (OPh)2 are shown. n shows the integer of 3-10000. Ph is the same as the above. At least one sort of phosphazene compounds chosen from the group which consists of straight chain-like phenoxy phosphazene expressed with ] are ophenylene group, m-phenylene group, p-phenylene group, and a general formula (3). [0029]

[Formula 8]

[0030] In the inside A of [type, -C(CH3)2-, -SO2-, -S-, or -O- is shown a shows 0 or 1. It is the compound over which at least one sort of bridge formation radicals chosen from the group which consists of a bis-phenylene group expressed with ] come to construct a bridge. (a) This bridge formation radical intervenes between two oxygen atoms from which the phenyl group of the phosphazene compound was desorbed. (b) The content ratio of a phenyl group can mention the bridge formation phenoxy phosphazene compound which does not have the hydroxyl group free to (c) intramolecular which is 50 to 99.9% on the basis of the total of the above-mentioned phosphazene compound (1) and/or all the phenyl groups in (2). In the above-mentioned general formula (2), the integer of n of 3-1000 is desirable. [0031] According to this invention, it is a general formula (4).

[0032]

[0033] It is the same as the above the inside m of [type. Annular dichlorophosphazene and the general formula (5) which are expressed with ]
[0034]
[Formula 10]

[0035] In the inside X2 of [type, radical-N=P[ N=PCl3 or radical-] (O) Cl is shown, and Y2 is a radical. - PCl4 or radical-P(O) Cl2 are shown. n is the same as the above. To at least one sort of dichlorophosphazene compounds chosen from the group which consists of straight chain-like dichlorophosphazene expressed with ], it is a general formula (6).

[0036]
[Formula 11]

[0037] In the inside M of [type, alkali metal is shown. The alkali-metal phenolate expressed with ], and a general formula (7) [0038]
[Formula 12]

[0039] It is the same as the above the inside M of [type. Alkali-metal JIFENORATO and the general formula (8) which are expressed with ]
[0040]
[Formula 13]

[0041] It is the same as the above the inside A, a, and M of [type. It is manufactured by making mixture with at least one sort of JIFENORATO chosen from the group which consists of alkali-metal JIFENORATO expressed with ] react, and making the above-mentioned alkali-metal phenolate react to the compound subsequently obtained further.

[0042] [namely, the bridge formation phenoxy phosphazene compound of this invention ] [at least one sort of dichlorophosphazene compounds chosen from the group which consists of straight chain-like dichlorophosphazene expressed with annular dichlorophosphazene and the general formula (5) which are expressed with the above-mentioned general formula (4) ]

Mixture with at least one sort of JIFENORATO chosen from the group which consists of an alkali-metal phenolate expressed with a general formula (6) and alkali-metal JIFENORATO expressed with alkali-metal JIFENORATO and the general formula (8) which are expressed with a general formula (7) is made to react (the first process). Subsequently, it is manufactured by what is made for the above-mentioned alkali-metal phenolate to react to the compound obtained further (the second process).

[0043] According to research of this invention person, it became clear that the reactivity over

the dichlorophosphazene compound of alkali-metal JIFENORATO expressed with a general formula (7) and a general formula (8) was extremely low compared with it of an alkali-metal phenolate which is expressed with a general formula (6). [ if mixed heating of the alkali-metal phenolate expressed with a dichlorophosphazene compound and a general formula (6) is more specifically carried out, the phenoxy phosphazene which the chlorine atom permuted by the phenoxy group will generate, but ] Even if it carries out mixed heating of alkali-metal JIFENORATO expressed with a dichlorophosphazene compound, a general formula (7), and/or a general formula (8), a substitution reaction hardly occurs. This is considered that the solubility to the solvent of alkali-metal JIFENORATO is extremely low, and dissociating thoroughly by the system of reaction has become a cause.

[0044] Therefore, for example, the method indicated to JP,S51-47042,A, namely, [ the method to which make an alkali-metal phenolate react to a dichlorophosphazene compound, and alkali-metal JIFENORATO is subsequently made to react ] It is dramatically difficult to permute thoroughly the chlorine atom which remains after a reaction with an alkali-metal phenolate by alkali-metal JIFENORATO. Even if one OM radical of alkali-metal JIFENORATO reacted with the chlorine atom in a dichlorophosphazene compound, OM radical of one end which remained will become difficult to react with other chlorine atoms, this OM radical will be changed into an OH radical, and the phosphazene compound with which the hydroxyl group remained as a result will generate.

[0045] On the other hand, according to this invention, the mixture of an alkali-metal phenolate and alkali-metal JIFENORATO is made to react to a dichlorophosphazene compound. Subsequently, by adopting the specific composition of making an alkali-metal phenolate react to the compound obtained further Without making a free hydroxyl group remain in intramolecular, M of both OM radical of alkali-metal JIFENORATO \*\*\*\*s, and the phenoxy (that is, molecular weight increased) phosphazene compound over which two oxygen atoms combined with the phosphorus atom in a dichlorophosphazene compound, and the bridge was constructed is manufactured.

[0046] [ the dichlorophosphazene compound expressed with the general formula (4) and general formula (5) which are used as one of the raw materials in the manufacturing method of this invention ] For example, according to the well-known method of a description, it can manufacture to JP,S57-87427,A, JP,S58-19604,B, JP,S61-1363,B, JP,S62-20124,B, etc. If the example is shown, it can manufacture by making an ammonium chloride and a phosphorus pentachloride (or an ammonium chloride, a phosphorus trichloride, and chlorine) react at about 120-130 degrees C, and carrying out demineralization oxidation in chlorobenzene, first. [0047] As an alkali-metal phenolate expressed with a general formula (6), a well-known thing can be used widely conventionally, for example, a sodium phenolate, a potassium phenolate, a lithium phenolate, etc. can be mentioned. These alkali-metal phenolate can use one sort independently, or can use two or more sorts together.

[0048] In alkali-metal JIFENORATO expressed with a general formula (7), two radical-OM (M is the same as the above) may be in alt.\*\* meta or which physical relationship of Para. As an example of this alkali-metal JIFENORATO, alkaline metal salt, such as resorcinol, hydroquinone, and a catechol, can be mentioned, for example. Also in these, sodium salt and lithium salt are desirable. This alkali-metal JIFENORATO can use one sort independently, or can use two or more sorts together.

[0049] As alkali-metal JIFENORATO expressed with a general formula (8) For example, alkaline metal salt, such as - oxydi phenol, and - sulfonyl diphenol (bisphenol S), and 4 and 4'-isopropylidene diphenol (bisphenol A), 4, and 4'4, 4'-thiodiphenol, 4, and 4'4, 4'-diphenol, etc. can be mentioned. Also in these, sodium salt and lithium salt are desirable. This alkalimetal JIFENORATO can use one sort independently, or can use two or more sorts together. [0050] In this invention, alkali-metal JIFENORATO expressed with alkali-metal JIFENORATO and the general formula (8) which are expressed with a general formula (7)

may be used independently, respectively, and these may be mixed and used.

[0051] In the first process of the manufacturing method of this invention so that the chlorine atom in a dichlorophosphazene compound may not be altogether consumed by a reaction with an alkali-metal phenolate and alkali-metal JIFENORATO That is, it is desirable to adjust the amount of an alkali-metal phenolate and the alkali-metal JIFENORATO used as the chlorine atom in a dichlorophosphazene compound remains in addition also by a reaction with an alkali-metal phenolate and alkali-metal JIFENORATO. Thereby, both-OM radical (M is the same as the above) of alkali-metal JIFENORATO combines with the phosphorus atom of a dichlorophosphazene compound. What is necessary is for about 0.05-0.9Eq of the amount of an alkali-metal phenolate and the alkali-metal JIFENORATO used just to be usually about 0.1-0.8Eq preferably with the total quantity of both phenolates on the basis of the chlorinity of a dichlorophosphazene compound at the first process.

[0052] In the second process of the manufacturing method of this invention, it is desirable to adjust the amount of the alkali-metal phenolate used so that all of all of the chlorine atom and the free hydroxyl group in the compound generated at the first process of the above may be consumed by a reaction with an alkali-metal phenolate. What is necessary is for about 1-1.5Eq of the amount of the alkali-metal phenolate used just to be usually about 1-1.2Eq preferably on the basis of the chlorinity of a dichlorophosphazene compound in this invention. [0053] this invention — the using rate (alkali-metal JIFENORATO / alkali-metal phenolate —) of an alkali-metal phenolate (total quantity used at the first process and the second process), and alkali-metal JIFENORATO The mole ratio should just usually set 2000-every/to 1 / 20 - 1/6 preferably 4 intensity.

[0054] the reaction of the first process and the second process -- each usual room temperature - it is preferably carried out under the temperature of about 80-140 degrees C, and about 150 degrees C is usually completed preferably in about 3 to 7 hours for about 1 to 12 hours. Each reaction of the first process and the second process is usually performed in organic solvents, such as halogenated-aromatics hydrocarbons, such as aromatic hydrocarbon, such as benzene, toluene, and a xylene, mono-chlorobenzene, and dichlorobenzene.

[0055] The bridge formation phenoxy phosphazene compound of this invention manufactured by the above-mentioned reaction follows the usual isolation methods, such as washing, filtration, and desiccation, and can be easily isolated and refined from a reaction mixture, for example.

[0056] The bridge formation phenoxy phosphazene compound of this invention is in within the limits whose decomposition temperature is 250-350 degrees C.

[0057] [moreover, the content ratio of the phenyl group in the bridge formation phenoxy phosphazene compound of this invention] It is 50 to 99.9% on the basis of the total of all the phenyl groups in the annular phenoxy phosphazene of a general formula (1), and/or the straight chain-like phenoxy phosphazene of a general formula (2), and is 70 to 90% preferably.

[0058] in addition, when the end groups X1 and Y1 in a general formula (2) change with reaction conditions etc., and are the usual reaction conditions and a mild reaction is performed, for example by the system of non-water X1 is set to -N=P(OPh) 3 and Y1 becomes the structure of -P(OPh)4. When it reacts by a severe reaction condition which is a reaction condition that moisture or alkali metal hydroxide exists in the system of reaction, or a transition reaction produces X1 will be in the state where X1 other than the structure of -P(OPh)4 is intermingled by -N=P(OPh) 3 and Y1, and the thing of the structure of -P(O) (OPh)2 is intermingled by -N=P(O) OPh and Y1.

[0059] The bridge formation phenoxy phosphazene compound of this invention is useful as a flame retarder.

[0060] [ the flame retarder which consists of a bridge formation phenoxy phosphazene compound of this invention ] Excluding [ therefore ] a halogen, it pyrolyzes at the time of

resin molding, and hydrogen halide is generated, and when the corrosion of metal mold, degradation of resin, and coloring are not caused and resin burns by a fire etc., harmful gas or smoke to a living thing, such as hydrogen halide, are not generated. Moreover, the bridge formation phenoxy phosphazene compound of this invention has low volatility, and does not reduce the molding temperature of resin, and does not have blocking or that ooze out (JUSHINGU) and problems, such as dripping at the time of combustion, arise at the time of kneading.

[0061] Furthermore, the flame retarder which consists of a bridge formation phenoxy phosphazene compound of this invention has not reducing molecular weight of resin and reducing [ little ] the properties of resin original, such as mechanical properties, such as shock resistance, thermal resistance, and fabricating-operation nature, by combination of a flame retarder.

[0062] [Triazine frame content compound] The triazine frame content compound which is the (C) component of this invention is a component (fire-resistant assistant) for using it with a phosphazene system flame retarder, and raising fire retardancy further.

[0063] In this invention, as long as it is the compound which contains the triazine frame in intramolecular as a triazine frame content compound, a well-known thing can be used widely conventionally. As an example of a triazine frame content compound For example, a melamine (the following type (9)), MERAMU (the following type (10)), MEREMU (the following type (11)), Although a melon (product of deammonia of MEREMU 3 to three molecules), a melamine SHIANU rate (the following type (12)), a phosphoric-acid melamine (the following type (13)), SAKUSHINO guanamine (the following type (14)), melamine resin (the following type (15)), etc. can be mentioned A melamine SHIANU rate is desirable especially from a viewpoint of volatility-proof and dispersibility.

[0064]

[Formula 14]

[0065] [Fire-resistant polyphenylene ether system resin composition object] [ the loadings of the bridge formation phosphazene system flame retarder of the (B) component blended into the fire-resistant polyphenylene ether system resin composition object of this invention ] (A) It is 1 - 30 weight section to the resin 100 weight section which consists of the polyphenylene ether resin 1 - 99 weight sections and the polystyrene system resin 99 - 1 weight section which are a component.

[0066] [moreover, the loadings of the triazine frame content compound of the (C) component blended into the fire-resistant polyphenylene ether system resin composition object of this invention ] (A) They are below 30 weight sections to the resin 100 weight section which consists of the polyphenylene ether resin 1 - 99 weight sections and the polystyrene system resin 99 - 1 weight section which are a component. [namely, the fire-resistant polyphenylene ether system resin composition object of this invention with which the triazine frame content compound of the (C) component was blended further ] (A) The resin 100 weight section which consists of the polyphenylene ether resin 1 - 99 weight sections and the polystyrene system resin 99 - 1 weight section which are a component, (B) The bridge formation phosphazene system flame retarder 1 of a component - 30 weight sections, and below the triazine frame content compound 30 weight section of the (C) component consist of the 0.1 - 30 weight section preferably.

[0067] [ especially the desirable presentation of the fire-resistant polyphenylene ether system resin composition object of this invention ] (A) The resin 100 weight section which consists of the polyphenylene ether resin 1 - 70 weight sections and the polystyrene system resin 99 -30 weight sections which are a component, (B) The bridge formation phosphazene system flame retarder 1 of a component - 30 weight sections, and below the triazine frame content compound 30 weight section of the (C) component are 0.1 - 30 weight section preferably. [0068] [Inorganic filler] An inorganic filler can be blended with the fire-resistant polyphenylene ether system resin composition object of this invention, in order to raise the mechanical physical properties of resin, and/or in order to raise dripping tightness further. [0069] As an inorganic filler, a well-known resin filling agent can be used widely conventionally. For example, a mica, a kaolin, talc, a silica, clay, a barium sulfate, A barium carbonate, a calcium carbonate, a calcium sulfate, a calcium silicate, Titanium oxide, a glass bead, a glass balloon, a glass flake, a glass fiber, Fibrous titanic-acid alkali metal (potassium titanate fiber etc.), fibrous boric-acid transition metal salt (boric-acid aluminum fiber etc.), Fibrous boric-acid alkaline earth metal salt (boric-acid magnesium fiber etc.), a zinc-oxide whisker, A titanium oxide whisker, a magnesium-oxide whisker, a gypsum-fibrosum whisker, An aluminum silicate (mineral name mullite) whisker, a calcium silicate (mineral name straw SUTONAITO) whisker, A silicon carbide whisker, a titanium carbide whisker, a silicon nitride whisker, a titanium nitride whisker, a carbon fiber, an alumina fiber, an alumina silica fiber, a zirconia fiber, a quartz fiber, etc. can be mentioned.

[0070] Also in these inorganic fillers, fibrous titanic-acid alkali metal, fibrous boric-acid transition metal salt, Fibrous boric-acid alkaline earth metal salt, a zinc-oxide whisker, a titanium oxide whisker, A magnesium-oxide whisker, an aluminum silicate whisker, a calcium silicate whisker, A silicon carbide whisker, a titanium carbide whisker, a silicon nitride whisker, What has shape anisotropy, such as fibrous material, such as a titanium nitride whisker, and a mica, is desirable, and fibrous titanic-acid alkali metal, fibrous boric-acid transition metal salt, fibrous boric-acid alkaline earth metal salt, a titanium oxide whisker, especially a calcium silicate whisker, etc. are desirable.

[0071] One sort can be independently used for these inorganic fillers, or they can use two or more sorts together.

[0072] Also in these inorganic bulking agents, what has shape anisotropy, such as whiskers and a mica, can be used preferably.

[0073] As the blending ratio of coal of an inorganic filler to polyphenylene ether system resin When the balance of improvement in mechanical physical properties and improvement in the fire-resistant engine performance is taken into consideration, it is [below 50 weight sections] usually good, although not limited in particular per polyphenylene ether system resin 100 weight section to consider it as 1 - 50 weight section preferably.

[0074] [Organic phosphorous compound which does not contain a halogen] In order to raise the fire retardancy further, the organic phosphorous compound (it is called the following "halogen free organic phosphorous compound") which does not contain a halogen can also be blended with the fire-resistant polyphenylene ether system resin composition object of this invention.

[0075] It is well-known that a halogen free organic phosphorous compound raises the fire retardancy of the matrix of resin etc. conventionally. However, the synergistic effect was discovered and this invention person found out that fire-resistant effectiveness was heightened remarkably, when the bridge formation phosphazene system flame retarder and halogen free organic phosphorous compound which are used in this invention were used together. [0076] As a halogen free organic phosphorous compound, a well-known thing can be used widely conventionally. For example, the thing of a description can be mentioned in JP,H6-19003,B, JP,H2-115262,A, JP,H5-1079,A, JP,H6-322277,A, a U.S. Pat. No. 5122556 Description, etc.

[0077] More specifically, for example Trimethyl phosphate, triethyl phosphate, Tributyl phosphate, trioctylphosphate, triphenyl phosphate, Tricresyl phosphate, trixylyl phosphate, cresyl diphenyl phosphate, Xylyl diphenyl phosphate, bird RUJIKI silyl phosphate, Phosphoric ester, such as tris (noryl phenyl) phosphate and diphenyl (2-ethylhexyl) phosphate, Hydroxyl-group content phosphoric ester, such as resorcinol diphenyl phosphate and hydroquinone diphenyl phosphate, 1, 3-phenylene screw (2, 6-dixylenyl phosphate), A resorcinol screw (diphenyl phosphate), a hydroquinone screw (diphenyl phosphate), The bisphenol A screw (diphenyl phosphate), a bisphenol S screw (diphenyl phosphate), A resorcinol screw (JIKISHIRIRU phosphate), a hydroquinone screw (JIKISHIRIRU phosphate), the bisphenol A screw (JITORIRU phosphate), a biphenol A screw (JIKISHIRIRU phosphate), Condensed-phosphoric-acid ester compounds, such as a bisphenol S screw (JIKISHIRIRU phosphate), A phosphine or phosphine oxide compounds, such as a bird lauryl phosphine, triphenyl phosphine, a tritolyl phosphine, triphenylphosphine oxide, and tritolyl phosphine oxide, etc. can be mentioned.

[0078] Also in a these halogens free organic phosphorous compound, triphenyl phosphate, Tricresyl phosphate, trixylyl phosphate, 1, 3-phenylene screw (2, 6-dixylenyl phosphate), A resorcinol screw (diphenyl phosphate), a hydroquinone screw (diphenyl phosphate), The bisphenol A screw (diphenyl phosphate), a resorcinol screw (JIKISHIRIRU phosphate), Condensed-phosphoric-acid ester compounds, such as a hydroquinone screw (JIKISHIRIRU phosphate) and the bisphenol A screw (JITORIRU phosphate), Phosphine oxide compounds, such as triphenylphosphine oxide and tritolyl phosphine oxide, etc. are desirable. Triphenyl phosphate, 1, 3-phenylene screw (2, 6-dixylenyl phosphate), a resorcinol screw (diphenyl phosphate), a resorcinol screw (JIKISHIRIRU phosphate), triphenylphosphine oxide, etc. are especially desirable.

[0079] One sort can be independently used for a halogen free organic phosphorous compound, or it can use two or more sorts together.

[0080] As the blending ratio of coal of a halogen free organic phosphorous compound to polyphenylene ether system resin When the balance of improvement in mechanical physical properties and improvement in the fire-resistant engine performance is taken into consideration, it is [below 50 weight sections] usually preferably good [below 30 weight sections], although not limited in particular per polyphenylene ether system resin 100 weight section to consider it as 1 - 30 weight section more preferably.

[0081] [Various additives] [ an object ] although the fire-resistant polyphenylene ether system resin composition object of this invention is a resin composition object which discovers the outstanding fire-resistant effectiveness without using the compound containing halogens, such as chlorine and a bromine, as a flameproofing component Usually, the additive for well-known flameproofing used can be suitably combined in the range which does not spoil the outstanding effectiveness, and it can also add.

[0082] If the additive for flameproofing usually discovers the flameproofing effectiveness, there will be no limit in particular. Metallic oxides, such as a zinc oxide, tin oxide, an iron oxide, molybdenum oxide, copper oxide, and a manganese dioxide, An aluminum hydroxide, a magnesium hydroxide, zirconium hydroxide, the aluminum hydroxide that carried out oxalic acid processing, Metal hydroxides, such as a magnesium hydroxide processed with the nickel compound, a sodium carbonate, Alkaline metal salt or alkaline earth metal salt, such as a calcium carbonate, a barium carbonate, and alkyl sulfonic-acid sodium, Chlorinated paraffin, par chloro cyclo pentadecane, tetrabromobisphenol A, An organochlorine compound or organic bromine compounds, such as an epoxy resin, bis(tribromo phenoxy) ethane, and bis(tetra-bromine FUTARU imino) ethane, Antimony compounds, such as an antimony trioxide, antimony tetroxide, antimony pentoxide, and sodium antimonate, Nitrogen containing compounds, such as red phosphorus, a halogen content phosphoric ester compound, a halogen content condensed-phosphoric-acid ester compound or a phosphonate compound, guanidine sulfamate, an ammonium sulfate, ammonium phosphate, ammonium polyphosphate, and alkylamine phosphate, zinc borate, The graphite of silicon compounds, such as boron compounds, such as metaboric acid barium and ammonium borate, silicone polymer, and a silica, and heat expansibility etc. can be mentioned.

[0083] One sort can be independently used for the additive for these flameproofing, or it can

use two or more sorts together.

[0084] Furthermore, it can blend with this invention fire retardancy polyphenylene ether system resin composition object from the former in the range which does not spoil the outstanding property, combining various well-known resin additives suitably. As a resin additive, for example Drip inhibitors, such as flame retarders other than the above, and polytetrafluoroethylene resin (PTFE) (dropping inhibitor), An ultraviolet ray absorbent, light stabilizer, an antioxidant, a shielding agent, metal deactivator, a quencher, a heat-resistant stabilizer, lubricant, a release agent, a coloring agent, an antistatic agent, an antioxidant, a plasticizer, an impact strength improvement agent, a compatibilizer, etc. can be mentioned. Moreover, you may blend various well-known additives further conventionally in order to give fog resistance, fungus resistance, antibacterial properties, or other functionality. [0085] [Manufacture of this invention flame-retardant-resin constituent] [ the fire-resistant polyphenylene ether system resin composition object of this invention ] To polyphenylene ether system resin, the above-mentioned bridge formation phosphazene system flame retarder and if needed A triazine frame content compound, Weighing capacity of the specified quantity of the various additives for an inorganic filler, a halogen free organic phosphorous compound, and flameproofing and other additives or the optimum dose can be carried out, and it can add, and can obtain by mixing and kneading by a well-known method. For example, the resin composition object of this invention can be obtained by kneading powder, a bead, a flake, or the mixture of each component of a pellet type using kneading machines, such as extruders, such as I shaft extruder and a biaxial extruder, a Banbury mixer, a pressurized kneader, and 2 rolls, etc. Moreover, when there is the need of blending a liquid, it can knead with an above-mentioned extruder or an above-mentioned kneading machine etc. using well-known liquid injection equipment.

[0086] [This invention flame-retardant-resin Plastic solid] By fabricating the fire-resistant polyphenylene ether system resin composition object of this invention, a fire-resistant polyphenylene ether system resin Plastic solid can be acquired. For example, of course, the

extrusion-molding article of various configurations, such as a resin sheet, a sheet, a film, and a section shape, can be manufactured from conventionally well-known shaping means, such as press forming, injection molding, and extrusion molding, and it is also possible to manufacture the resin sheet of a bilayer or three layer systems using a co-extrusion kneading machine etc.

[0087] [ thus, the fire-resistant polyphenylene ether system resin composition object of this invention and the fire-resistant polyphenylene ether system resin Plastic solid which are acquired ] It can be used for the broad industrial fields, such as the electrical and electric equipment, an electron and communication, agriculture-and-forestry fishery, mining, construction, food, fiber, clothing, Medical Science Division, coal, petroleum, rubber, leather, an automobile, a precision mechanical equipment, timber, furniture, printing, and a musical instrument.

[0088] More specifically [ the fire-resistant polyphenylene ether system resin composition object of this invention, and a fire-resistant polyphenylene ether system resin Plastic solid ] A printer, a personal computer, a word processor, a keyboard, PDA (small information terminal), Telephone, facsimile, a copying machine, ECR (electronic cash register), a calculator, Clerical work and OA equipment, such as an electronic notebook, an electronic dictionary, a card, an electrode holder, and stationery, A washer, a refrigerator, a cleaner, a microwave oven, lighting fitting, a game machine, an iron, Electrical household appliances and electrical equipment, such as a kotatsu, TV and VTR, a video camera, a radio cassette recorder, a tape recorder, AV equipment, such as a mini disc, CD player, a loudspeaker, and a liquid crystal display, It is used for the application of components, such as the electrical and electric equipment and electronic parts, such as a connector, a relay, a capacitor, a switch, a printed circuit board, a coil bobbin, a semi-conductor closure ingredient, an electric wire, a cable, a transformer, a deflecting yoke, a panelboard, and a clock, and communication equipment, an automobile, a car, a marine vessel, and an aircraft, a structural ingredient, etc. [0089]

[Example] A synthetic example, a work example, a comparative example, and the example of reference are given to below, and this invention is explained in more detail. That it is in below with the "section" and "%" means the "weight section" and "weight %", respectively. Moreover, that it is with "-Ph" and "-Ph -" means a "phenyl group" and a "phenylene group", respectively. Furthermore, various inner assessment, such as a work example, is the values measured as follows.

[0090] 1. Heat deflection temperature: ASTM According to D-648, it measured by load 18.6 kgf/cm2 and was considered as the heat-resistant index.

[0091] Fire Retardancy: 2. It is Based on Method (Test for Flammability of Plastic Materials for Parts in Devices and Appliances UL94, Fourth Edition) of Examining UL-94. A test piece 1/16 inch in thickness, 5 inches in die length, and 0.5 inch in width It used and the evaluation test was carried out. A valuation basis is shown below.

V-0: Fulfill all the conditions of following (A) - (E).

- (A) Flaming of every one-set five test piece after an approaching flame (flame is raised and it continues burning) is 10 or less seconds.
- (B) The flaming sum total after a total of every 2 times ten approaching flames is less than 50 seconds to one-set five test pieces.
- (C) Carry out flaming of no the one-set five test pieces to a clamp.
- (D) No one-set five test pieces trickle the flaming grain which ignites the cotton under 305mm.
- (E) After the 2nd approaching flame and glowing (although flame is raised and it does not burn, it remains as red-hot charcoal) of every one-set five test piece are less than 30 seconds. V-1: Fulfill all the conditions of following (A) (E).
- (A) Flaming of every one-set five test piece after an approaching flame is less than 30

seconds.

(B) The flaming sum total after a total of every 2 times ten approaching flames is less than 250 seconds to one-set five test pieces.

(C) And (D) is the same as V-0.

(E) After the 2nd approaching flame and glowing of every one-set five test piece are less than 60 seconds.

V-2: Fulfill all the conditions of following (A) - (E).

(A), (B), (C), and (E) are the same as V-1.

(D) One or more of one-set five test pieces trickle the flaming grain which ignites the cotton under 305mm.

At least one is not given to the conditions of NG:V-2.

[0092] 3. Volatile gas and discoloration at the time of molding: it checked by viewing. [0093] Polyphenylene ether, polystyrene system resin, the halogen free organic phosphorous compound, and the inorganic filler used the following.

Polyphenylene ether (PPE): Pori (2, the 6-dimethyl 1, 4-phenylene ether), [limiting viscosity eta] 0.56 dl/g.

High-impact-polystyrene (HIPS): (trade name: HIPS H550, product made from Japanese Polystyrene)

Polystyrene (GPPS): (trade name: GPPS G690N, product made from Japanese Polystyrene) Aromatic series phosphoric ester: (triphenyl phosphate (TPP), Wako Pure Chem make) Aromatic-series condensed-phosphoric-acid ester: 1, 3-phenylene screw (2, 6-dixylenyl phosphate) (trade name PX-200, product made from Daihachi Chemical industry) Inorganic filler: Potassium titanate fiber (trade name: TISMO-D N, product made from Otsuka Chemistry)

Straw SUTONAITO (trade name: by SUTARUW, product made from Otsuka Chemistry). [0094] The synthetic example 1 (composition of the phenoxy phosphazene compound which has the structure of cross linkage by p-phenylene group)

The toluene solution of the sodium phenolate was prepared by carrying out the heating reflux of Phenol 103.5g (1.1mol), 44.0g (1.1mol) of sodium hydroxides, 50g of water, and the toluene 500ml mixture, and removing only water besides a system.

[0095] It is concurrent with said reaction and is Hydroquinone 16.5g (0.15mol) to a 21.4 Thu opening flask, The toluene solution of lithium salt of hydroquinone and a phenol was prepared by putting in Phenol 94.1g (1.0mol), 31.1g (1.3mol) of lithium-hydroxides, 52g of water, and toluene 600ml, carrying out heating reflux, and removing only water besides a system. To this toluene solution, it is a dichlorophosphazene oligomer (62% of trimer). After 580g of 20% chlorobenzene solutions as for which 12% of a tetramer, a pentamer and 11% of a hexamer, 3% of the amount object of seven, and more than an octamer contain a 12% of mixture 1.0 unit mol (115.9g) were dropped below 30 degrees C under churning, the churning reaction of them was carried out at 110 degrees C for 3 hours. Next, after adding under churning the toluene solution of a sodium phenolate prepared previously, the reaction was continued at 110 degrees C for 4 hours.

[0096] After 1.01, of sodium-hydroxide water solutions washed the reaction mixture 3 times 3% after termination of a reaction, next 1.01. of water washed 3 times, the organic layer was condensed under reduced pressure. The heating vacuum drying of the obtained product was carried out by 80 degrees C and 3 mmHg or less for 11 hours, and 211g of fine yellow powder of bridge formation phenoxy phosphazene was obtained.

[0097] Bridge formation phenoxy phosphazene obtained above, It became clear that the presentation by 0.04% of hydrolysis chlorine, the weight average molecular weight (Mw) 1100 (polystyrene conversion, GPC analysis), phosphorus content, and a CHN elementalanalysis value was [N=P(-O-p-Ph-O-) 0.15(-O-Ph) 1.7] mostly.

[0098] TG/DTA analysis (thermogravimetric analysis) did not show the clear melting point,

but 306 degrees C and the 5% weight loss temperature of decomposition starting temperature were 311 degrees C.

[0099] Moreover, as a result of performing the quantum of a residual hydroxyl group by the acetylating method, it was below limit of detection (considering it as the hydroxyl equivalent per sample 1g:1x10-6Eq/(g)). In addition, limit of detection is a same number value also in the following synthetic examples.

[0100] The synthetic example 2 (composition of the phenoxy phosphazene compound which has the structure of cross linkage by a 2 and 2-bis(p-oxy-phenyl) isopropylidene radical). Phenol 65.9g (0.7mol) and toluene 500ml was put into the 11.4 Thu opening flask, and under churning, keeping internal solution temperature at 25 degrees C, 14.9g of metallic-sodium 0.65 gram atoms were cut out finely, and were thrown in. Churning was continued for 8 hours until metallic sodium disappeared thoroughly at 77-113 degrees C after termination of the charge, and the sodium-phenolate solution was prepared.

[0101] It is concurrent with said reaction and bisphenol A. 57.1g (0.25mol), Phenol 103.5g (1.1mol) and tetrahydrofuran (THF) 800ml was put into the 31.4 Thu opening flask, and under churning, keeping internal solution temperature at 25 degrees C or less, 11.1g of metal lithium 1.6 gram atoms were cut out finely, and were thrown in. Churning was continued for 8 hours after termination of the charge until metal lithium disappeared thoroughly at 61-68 degrees C. It is a dichlorophosphazene oligomer (concentration: 37%) to this slurry solution. 313g of chlorobenzene solutions, a presentation: Under churning of a 1% of mixture 1.0 unit mol (115.9g), 75% of a trimer, 17% of a tetramer, 5 and 6% of the hexamer, 1% of the amount object of seven, and more than the octamer reacted at 80 degrees C for 2 hours, after being dropped over 1 hour, keeping internal solution temperature at 20 degrees C or less. Subsequently, under churning, after adding the sodium-phenolate solution prepared separately over 1 hour, keeping internal solution temperature at 20 degrees C, the reaction was carried out at 80 degrees C for 5 hours.

[0102] After termination of a reaction, the reaction mixture was condensed and 11. of toluene was newly added except for THF. It is 2%NaOH about this toluene solution. After washing, next 1.01. of water washed 3 times 3 times at 11., the organic layer was condensed under reduced pressure. The heating vacuum drying of the obtained product was carried out by 80 degrees C and 3 mmHg or less for 11 hours, and 229g of white powder of bridge formation phenoxy phosphazene was obtained.

[0103] Bridge formation phenoxy phosphazene obtained above, 0.07% of hydrolysis chlorine, the weight average molecular weight (Mw) 1130 (polystyrene conversion, GPC analysis), phosphorus content, and a CHN elemental-analysis value \*\*\*\* presentation were [N=P(-O-Ph-C(CH3) 2-Ph-O-) 0.25(-O-Ph) 1.50].

[0104] TG/DTA analysis did not show the clear melting point, but 308 degrees C and the 5% weight loss temperature of decomposition starting temperature were 313 degrees C. Moreover, the amount of residual hydroxyl groups was below limit of detection (the acetylating method).

[0105] The synthetic example 3 (composition of the phenoxy phosphazene compound which has the structure of cross linkage by a meta-phenylene group)

It replaced with hydroquinone, and using resorcinol, reaction and processing were performed like the synthetic example 1, and 209g of white powder of bridge formation phenoxy phosphazene was obtained.

[0106] 0.08% of hydrolysis chlorine, the weight average molecular weight (Mw) 1080 (polystyrene conversion, GPC analysis), phosphorus content, and the CHN elemental-analysis value \*\*\*\* presentation of the bridge formation phenoxy phosphazene obtained above were [N=P(-O-m-Ph-O-) 0.15(-O-Ph) 1.7].

[0107] TG/DTA analysis did not show the clear melting point, but 304 degrees C and the 5% weight loss temperature of decomposition starting temperature were 311 degrees C.

Moreover, the amount of residual hydroxyl groups was below limit of detection (the

acetylating method).

[0108] The synthetic example 4 (composition of the phenoxy phosphazene which has the structure of cross linkage by a 4 and 4'-sulfonyl diphenylene radical (bisphenol S residue)) Phenol 103.5g (1.1mol) and THF 500ml was put into the 11. 4 Thu opening flask, and under churning, keeping internal solution temperature at 25 degrees C, 25.3g of metallic-sodium 1.1 gram atoms were cut out finely, and were thrown in. Churning was continued for 5 hours until metallic sodium disappeared thoroughly at 65-72 degrees C after termination of the charge. [0109] It is concurrent with said reaction and they are Phenol 94.1g (1.0mol) and Bisphenol S with a 11. 4 opening flask. It is THF about 6.26g (0.025mol). It dissolves in 500ml. 24.1g of debris of metallic-sodium 1.05 gram atom is supplied below 25 degrees C, and it applies after termination of the charge for 1 hour, and is 61 degrees C. Churning was continued at temperature up and 61-68 degrees C for 6 hours, and the sodium-phenolate mixed solution was prepared. This solution Dichlorophosphazene oligomer ([ 62% of a trimer, 12% of a tetramer, a pentamer, and 11% of hexamer ]) The chuming reaction was carried out at 71-73 degrees C after dropping under cooling / churning of 25 degrees C or less for 5 hours at 580g of 20% chlorobenzene solutions as for which 3% of the amount object of seven and more than an octamer contain a 12% of mixture 1.0 unit mol (115.9g). Next, after the sodium-phenolate solution prepared previously was dropped, the reaction was continued at 71-73 degrees C for 3 hours, and the sodium phenolate was prepared.

[0110] After condensing the reaction mixture after termination of a reaction and remelting to chlorobenzene 500ml, 3 times, 5% sulfuric acid treatment, 5% sodium bicarbonate backwashing by water, and three rinsings were performed, concentration hardening by drying of the 5%NaOH backwashing by water was carried out, and 216g of wax-like objects of the straw color of bridge formation phenoxy phosphazene were obtained.

[0111] The bridge formation phenoxy phosphazene obtained above is 0.05% of hydrolysis chlorine, and the weight average molecular weight (Mw) 1030 (polystyrene conversion). The presentation was mostly determined as [N=P(-O-Ph-SO2-Ph-O-) 0.025(-O-Ph) 1.95] with phosphorus content and a CHN elemental-analysis value.

TG/DTA analysis; melting out temperature Tm:103 degree C, decomposition starting temperature:321 degree C, and below amount of 5% weight loss temperature:332-degree-C residual hydroxyl groups: limit of detection (the acetylating method)

[0112] The synthetic example 5 (composition of the phenoxy phosphazene which has the structure of cross linkage by a 4 and 4'-sulfonyl diphenylene radical (bisphenol S residue)) The sodium phenolate was prepared like the synthetic example 4 using Phenol 37.6g (0.4mol) and 9.2g of metallic-sodium 0.4 gram atoms.

[0113] Moreover, Phenol 160.0g (1.70mol), Bisphenol S The mixture of a sodium phenolate and a NATORIUMUII phenolate was prepared like the synthetic example 4 using 12.5g (0.05mol) and metallic-sodium 1.8 gram atom (41.4g). This mixture Dichlorophosphazene oligomer ([62% of a trimer, and 12% of tetramer]) [580g of 20% chlorobenzene solutions as for which a pentamer and 11% of a hexamer, 3% of the amount object of seven, and more than an octamer contain a 12% of mixture 1.0 unit mol (115.9g)] It was dropped under cooling / churning of 25 degrees C or less, and was operated like the synthetic example 4 below, 218g of straw color wax-like objects of bridge formation phenoxy phosphazene were obtained, and it checked that they were the following compounds as a result of analysis. [N=P(-O-Ph-SO2-Ph-O-)0.05(-O-Ph)1.90]

residual chlorine: - 0.01% or less weight-average-molecular-weight Mw:1080 (polystyrene conversion)

TG/DTA analysis; melting out temperature Tm:103 degree C, decomposition starting temperature:320 degree C, and below amount of 5% weight loss temperature:334-degree-C residual hydroxyl groups:limit of detection

[0114] The synthetic example 6 (composition of the phenoxy phosphazene which has the structure of cross linkage by a 4 and 4'-sulfonyl diphenylene radical (bisphenol S residue)) The sodium phenolate was prepared like the synthetic example 4 using Phenol 37.6g (0.4mol) and 9.2g of metallic-sodium 0.4 gram atoms.

[0115] Moreover, Phenol 141.2g (1.50mol), Bisphenol S The mixture of a sodium phenolate and a NATORIUMUJI phenolate was prepared like the synthetic example 4 using 37.5g (0.15mol) and 41.4g of metallic-sodium 1.8 gram atoms. This mixture Dichlorophosphazene oligomer ([62% of a trimer, and 12% of tetramer]) [580g of 20% chlorobenzene solutions as for which a pentamer and 11% of a hexamer, 3% of the amount object of seven, and more than an octamer contain a 12% of mixture 1.0 unit mol (115.9g) ] It was dropped under cooling / churning of 25 degrees C or less, and was operated like the synthetic example 4 below, 217g of straw color wax-like objects of bridge formation phenoxy phosphazene were obtained, and it checked that they were the following compounds as a result of analysis. [N=P(-O-Ph-SO2-Ph-O-)0.15(-O-Ph)1.70]

residual chlorine: -- 0.03% weight-average-molecular-weight Mw:1150 (polystyrene conversion)

TG/DTA analysis; melting out temperature Tm:detection improper, decomposition starting temperature:318 degree C, and below amount of 5% weight loss temperature:335-degree-C residual hydroxyl groups (acetylating method):limit of detection

[0116] The synthetic example 7 (composition of the phenoxy phosphazene which has the structure of cross linkage by a 4 and 4'-oxydiphenylene radical)

The sodium phenolate was prepared like the synthetic example 4 using Phenol 94.1g (1.0mol) and 23.0g of metallic-sodium 1.0 gram atoms.

[0117] Moreover, the mixture of a sodium phenolate and a NATORIUMUJI phenolate was prepared like the synthetic example 4 using Phenol 94.1g (1.0mol), the bis(4-hydroxyphenyl) ether 20.2g (0.10mol), and 27.6g of metallic-sodium 1.2 gram atoms. This mixture Dichlorophosphazene oligomer ([62% of a trimer, and 12% of tetramer]) [580g of 20% chlorobenzene solutions as for which a pentamer and 11% of a hexamer, 3% of the amount object of seven, and more than an octamer contain a 12% of mixture 1.0 unit mol (115.9g) ] It was dropped under cooling / churning of 25 degrees C or less, and was operated like the synthetic example 4 below, 211g of straw color wax-like objects of bridge formation phenoxy phosphazene were obtained, and it checked that they were the following compounds as a result of analysis.

[0118] [N=P(-O-Ph-O-Ph-O-)0.1(-O-Ph)1.8]

Residual chlorine: 0.01% or less weight-average-molecular-weight Mw:1100 (polystyrene conversion)

TG/DTA analysis; melting out temperature Tm:detection improper, decomposition starting temperature:321 degree C, and below amount of 5% weight loss temperature:328-degree-C residual hydroxyl groups (acetylating method):limit of detection

[0119] The synthetic example 8 (composition of the phenoxy phosphazene which has the structure of cross linkage by a 4 and 4'-oxydiphenylene radical)

The sodium phenolate was prepared like the synthetic example 4 using Phenol 94.1g (1.0mol) and 27.6g of metallic-sodium 1.2 gram atoms.

[0120] Moreover, the mixture of a sodium phenolate and a NATORIUMUII phenolate was prepared like the synthetic example 4 using Phenol 94.1g (1.0mol), the bis(4-hydroxyphenyl) ether 40.4g (0.20mol), and 27.6g of metallic-sodium 1.2 gram atoms. This mixture Dichlorophosphazene oligomer ([62% of a trimer, and 12% of tetramer]) [580g of 20% chlorobenzene solutions as for which a pentamer and 11% of a hexamer, 3% of the amount object of seven, and more than an octamer contain a 12% of mixture 1.0 unit mol (115.9g)] It was dropped under cooling / churning of 25 degrees C or less, and was operated like the synthetic example 4 below, 212g of straw color wax-like objects of bridge formation phenoxy

phosphazene were obtained, and it checked that they were the following compounds as a result of analysis.

[0121] [N=P(-O-Ph-O-Ph-O-)0.2(-O-Ph)1.6]

Residual chlorine: 0.02% weight-average-molecular-weight Mw:1220 (polystyrene conversion)

It can TG/DTA-analyze, and can melting-out-temperature-Tm, : cannot detect, but they are decomposition starting temperature:306 degree C and below amount of 5% weight loss temperature:321-degree-C residual hydroxyl groups (acetylating method):limit of detection. [0122] The synthetic example 9 (composition of the phenoxy phosphazene which has the structure of cross linkage by a 4 and 4'-thio diphenylene radical)

The sodium phenolate was prepared like the synthetic example 4 using Phenol 94.1g (1.0mol) and 23.0g of metallic-sodium 1.0 gram atoms.

[0123] Moreover, the mixture of a sodium phenolate and a NATORIUMUJI phenolate was prepared like the synthetic example 4 using phenols 94.1g (1.0mol) and 4, the 4'-thiodiphenol 21.8g (0.10mol), and 27.6g of metallic-sodium 1.2 gram atoms. This mixture Dichlorophosphazene oligomer ([62% of a trimer, and 12% of tetramer]) [580g of 20% chlorobenzene solutions as for which a pentamer and 11% of a hexamer, 3% of the amount object of seven, and more than an octamer contain a 12% of mixture 1.0 unit mol (115.9g) ] It was dropped under cooling / churning of 25 degrees C or less, and was operated like the synthetic example 4 below, 215g of straw color high viscosity objects of bridge formation phenoxy phosphazene were obtained, and it checked that they were the following compounds as a result of analysis.

[0124] [N=P(-O-Ph-S-Ph-O-)0.1(-O-Ph)1.8]

Residual chlorine: 0.07% weight-average-molecular-weight Mw:1210 (polystyrene conversion)

It can TG/DTA-analyze, and can melting-out-temperature-Tm, : cannot detect, but they are decomposition starting temperature:337 degree C and below amount of 5% weight loss temperature:342-degree-C residual hydroxyl groups (acetylating method):limit of detection. [0125] The synthetic example 10 (composition of the phenoxy phosphazene which has the structure of cross linkage by a 4 and 4'-thio diphenylene radical)

The sodium phenolate was prepared like the synthetic example 4 using Phenol 94.1g (1.0mol) and 23.0g of metallic-sodium 1.0 gram atoms.

[0126] Moreover, the mixture of a sodium phenolate and a NATORIUMUJI phenolate was prepared like the synthetic example 4 using phenols 94.1g (1.0mol) and 4, the 4'-thiodiphenol 43.7g (0.20mol), and 27.6g of metallic-sodium 1.2 gram atoms. This mixture Dichlorophosphazene oligomer ([ 62% of a trimer, and 12% of tetramer ]) [ 580g of 20% chlorobenzene solutions as for which a pentamer and 11% of a hexamer, 3% of the amount object of seven, and more than an octamer contain a 12% of mixture 1.0 unit mol (115.9g) ] It was dropped under cooling / churning of 25 degrees C or less, and was operated like the synthetic example 4 below, 217g of straw color high viscosity objects of bridge formation phenoxy phosphazene were obtained, and it checked that they were the following compounds as a result of analysis.

[0127] [N=P(-O-Ph-S-Ph-O-)0.2(-O-Ph)1.6]

Residual chlorine: 0.01% weight-average-molecular-weight Mw:1320 (polystyrene conversion)

It can TG/DTA-analyze, and can melting-out-temperature-Tm, : cannot detect, but they are decomposition starting temperature:341 degree C and below amount of 5% weight loss temperature:347-degree-C residual hydroxyl groups (acetylating method):limit of detection. [0128] The synthetic example 11 (composition of the phenoxy phosphazene which has the structure of cross linkage by a 4 and 4'-diphenylene radical)

The sodium phenolate was prepared like the synthetic example 4 using Phenol 94.1g (1.0mol)

and 23.0g of metallic-sodium 1.0 gram atoms.

[0129] Moreover, the mixture of a sodium phenolate and a NATORIUMUJI phenolate was prepared like the synthetic example 4 using phenols 94.1g (1.0mol) and 4, the 4'-diphenol 18.6g (0.10mol), and 27.6g of metallic-sodium 1.2 gram atoms. This mixture Dichlorophosphazene oligomer ([62% of a trimer, and 12% of tetramer]) [580g of 20% chlorobenzene solutions as for which a pentamer and 11% of a hexamer, 3% of the amount object of seven, and more than an octamer contain a 12% of mixture 1.0 unit mol (115.9g)] It was dropped under cooling / churning of 25 degrees C or less, and was operated like the synthetic example 4 below, 208g of straw color high viscosity objects of bridge formation phenoxy phosphazene were obtained, and it checked that they were the following compounds as a result of analysis.

[0130] [N=P(-O-Ph-Ph-O-)0.1(-O-Ph)1.8]

Residual chlorine: 0.01% weight-average-molecular-weight Mw:1210 (polystyrene conversion)

It can TG/DTA-analyze, and can melting-out-temperature-Tm,: cannot detect, but they are decomposition starting temperature:338 degree C and below amount of 5% weight loss temperature:349-degree-C residual hydroxyl groups (acetylating method):limit of detection. [0131] The synthetic example 12 (composition of the phenoxy phosphazene which has the structure of cross linkage by a 4 and 4'-diphenylene radical)

The sodium phenolate was prepared like the synthetic example 4 using Phenol 94.1g (1.0mol) and 23.0g of metallic-sodium 1.0 gram atoms.

[0132] Moreover, the mixture of a sodium phenolate and a NATORIUMUJI phenolate was prepared like the synthetic example 4 using phenols 94.1g (1.0mol) and 4, the 4'-diphenol 37.2g (0.20mol), and 27.6g of metallic-sodium 1.2 gram atoms. This mixture Dichlorophosphazene oligomer ([62% of a trimer, and 12% of tetramer]) [580g of 20% chlorobenzene solutions as for which a pentamer and 11% of a hexamer, 3% of the amount object of seven, and more than an octamer contain a 12% of mixture 1.0 unit mol (115.9g)] It was dropped under cooling / churning of 25 degrees C or less, and was operated like the synthetic example 4 below, 211g of straw color high viscosity objects of bridge formation phenoxy phosphazene were obtained, and it checked that they were the following compounds as a result of analysis.

[0133] [N=P(-O-Ph-Ph-O-)0.2(-O-Ph)1.6]

Residual chlorine: 0.01% weight-average-molecular-weight Mw:1350 (polystyrene conversion)

It can TG/DTA-analyze, and can melting-out-temperature-Tm,: cannot detect, but they are decomposition starting temperature:336 degree C and below amount of 5% weight loss temperature:347-degree-C residual hydroxyl groups (acetylating method):limit of detection. [0134] It is a flameproofing agent to the resinous principle 100 weight section which consists of 80 copies of work-example 1 Pori (2, the 6-dimethyl 1, 4-phenylene ether), ten copies of high impact polystyrene (HIPS), and ten copies of polystyrene (GPPS). Five copies of bridge formation phenoxy phosphazene compounds manufactured in the synthetic example 1 are added, and they are after mixing and 25mm with a mixer. Fusion kneading was carried out using the biaxial kneading machine, and the flame-retardant-resin constituent was obtained. [0135] The 1/16-inch-thick test piece was produced for this constituent with injection molding, and heat deflection temperature was measured according to D-648 of the fire-resistant assessment based on the method of examining UL-94, and ASTM. As a result, fire retardancy is V-0, heat deflection temperature is 150 degrees C, and JUSHINGU was not accepted at the time of molding.

[0136] The flame-retardant-resin constituent was obtained like the work example 1 except using the bridge formation phenoxy phosphazene compound manufactured in a work example 2 - the examples 2-12 of 12 composition.

[0137] The flame-retardant-resin constituent was obtained like the work example 1 except adding a work example 13 and also 15 copies of straw SUTONAITO.

[0138] Replace with the bridge formation phenoxy phosphazene compound manufactured in the example 1 of work-example 14 composition, and make it be the same as that of a work example 1 except using one copy of three copies of bridge formation phenoxy phosphazene compound [ which were manufactured in the synthetic example 2 ] and 1, and 3-phenylene screw (2, 6-dixylenyl phosphate) (PX-200). The flame-retardant-resin constituent was obtained.

[0139] It replaces with the bridge formation phenoxy phosphazene compound manufactured in the comparative example 1 - the example 1 of 3 composition, and is TPP. Five copies (comparative example 1) or PX-200 The flame-retardant-resin constituent was obtained like the work example 1 except blending five copies (comparative example 2), or not blending a flame retarder (comparative example 3).

[0140] Production and assessment of the test piece were performed like the work example 1 using the flame-retardant-resin constituent obtained by the flame-retardant-resin constituent and comparative examples 1-3 which are acquired in the above-mentioned work examples 2-14. These results are shown in a table 1.

[0141] In addition, as for the resin composition object of the comparative example 1, JUSHINGU was accepted at the time of molding. Moreover, the resin composition object of the comparative example 3 did not show fire retardancy at all.

[0142]

| com and | -8  | ** |
|---------|-----|----|
| Table   | 1   |    |
| 1 minu  | - 5 |    |
|         |     |    |

|              |    |      |           |           |      | des 155 143 |
|--------------|----|------|-----------|-----------|------|-------------|
|              |    | 樹脂成分 | 架橋ホスファ    |           | 熱変形  | 難燃性         |
|              |    |      | ゼン化合物     | 無微質充填剂    | 温度   | 試験          |
|              |    | (88) | (部)       | (部)       | (°C) | UL-94       |
|              | 1  | 100  | 合成例1:5    | _         | 150  | V-0         |
|              | 2  | 100  | 合成例2:5    | Name to   | 151  | V0          |
|              | 3  | 100  | 合成例3:5    |           | 151  | V-0         |
| 寒            | 4  | 100  | 合成例4:5    |           | 150  | V-0         |
|              | 5  | 100  | 合成例 5 : 5 |           | 151  | V0          |
|              | ຕິ | 100  | 合成例 6:5   |           | 151  | V-0         |
|              | 7  | 100  | 合成例7:5    |           | 151  | V0          |
| 施            | 8  | 100  | 合成例8:5    |           | 150  | V-0         |
|              | 9  | 100  | 合成例9;5    |           | 150  | V-0         |
|              | 10 | 100  | 合成倒10:5   |           | 151  | V-0         |
|              | 11 | 100  | 合成例 11:5  |           | 150  | V-0         |
| <i>\$</i> 58 | 12 | 100  | 合成例 12:5  |           | 151  | V-0         |
|              | 13 | 100  | 合成例 1:5   | バイスタルW:15 | 150  | V-0         |
|              | 14 | 100  | 合放例2:3    | PX-200:1  | 148  | V-0         |
| 比            | 1  | 100  |           | TPP:5     | 128  | V-0         |
| 較            | 2  | 100  | -         | PX-200:5  | 138  | V-0         |
| Ø            | 3  | 100  |           |           | 168  | NG          |

[0143] A resinous principle is the mixture of PPE/PS resin =80/20 among a table 1. Inorganic bulking agent and PX-200 and TPP of straw SUTONAITO are halogen free organic phosphorous compounds.

[0144] Work-example 15 Pori (as 15 copies of bridge formation phenoxy phosphazene compounds manufactured in the synthetic example 1 in the resinous principle 100 weight section which consists of 2, the 6-dimethyl 1, 50 copies of 4-phenylene ether, 25 copies of high impact polystyrene (HIPS), and 25 copies of polystyrene (GPPS), and a fire-resistant assistant) Ten copies of melamine SHIANU rates are added, and they are after mixing and

25mm with a mixer. Fusion kneading was carried out using the biaxial kneading machine, and the flame-retardant-resin constituent was obtained.

[0145] The 1/16-inch-thick test piece was produced for this constituent with injection molding, and heat deflection temperature was measured according to D-648 of the fire-resistant assessment based on the method of examining UL-94, and ASTM. As a result, fire retardancy is V-0, heat deflection temperature is 101 degrees C, and JUSHINGU was not accepted at the time of molding.

[0146] It replaced with the bridge formation phenoxy phosphazene compound manufactured in the work example 16 - the example 1 of 26 composition, and the flame-retardant-resin constituent was obtained like the work example 15 except using the bridge formation phenoxy phosphazene compound manufactured in the synthetic examples 2-12.

[0147] It replaced with the bridge formation phenoxy phosphazene compound manufactured in the example 1 of work-example 27 composition, the bridge formation phenoxy phosphazene compound manufactured in the synthetic example 3 was used, and the flame-retardant-resin constituent was obtained like the work example 15 except adding 15 copies of potassium titanate fibers (TISMO-D N) further.

[0148] Ten copies of bridge formation phenoxy phosphazene compounds which replaced with the bridge formation phenoxy phosphazene compound manufactured in the example 1 of work-example 28 composition, and were manufactured in the synthetic example 4, The flame-retardant-resin constituent was obtained like the work example 15 except using two copies of 1 and 3-phenylene screws (2, 6-dixylenyl phosphate) (PX-200).

[0149] It replaces with the bridge formation phenoxy phosphazene compound manufactured in the comparative example 4 - the example 1 of 7 composition. TPP 15 copies (comparative example 4) or PX-200 blending 15 copies (comparative example 5), or not blending only a flame retarder — and the loadings of a fire-resistant assistant — 20 copies — carrying out (comparative example 6) — or make it be the same as that of a work example 15 except not blending both a flame retarder and a fire-resistant assistant (comparative example 7) The flame-retardant-resin constituent was obtained.

[0150] Production and assessment of the test piece were performed like the work example 15 using the flame-retardant-resin constituent obtained by the flame-retardant-resin constituent and comparative examples 4-7 which are acquired in the above-mentioned work examples 16-28. These results are shown in a table 2.

[0151] In addition, as for the resin composition object of the comparative example 4, JUSHINGU was accepted at the time of molding. Moreover, the resin composition object of the comparative example 7 did not show fire retardancy at all.

[0152]

[Table 2]

|        | ۳    | Marine Phase  | #0-592 Jan 77 77 | 他の離燃剤、整燃助剤              | 效变形 | 雞然性      |
|--------|------|---------------|------------------|-------------------------|-----|----------|
|        |      | 191 HE IX 70" | 契橋ホスファ<br>ゼン化合物  | ソは経接質充填剤                | 湿度  |          |
|        |      | (部)           | (58)             | (部)                     | (%) | UL-94    |
| 7      | 15   |               | 合成例1:15          | <b>メラミン・シアヌレート 1 15</b> | 101 | V0       |
|        | 16   | 100           | 合成例2:15          | メラミン・シアヌレート:15          | 102 | V-0      |
|        | 17   | 100           | 合成例3:15          | メラミン・シアヌレート:15          | 101 | V-0      |
| 実      | 18   | 100           | 合成例4:15          | メラミン・シアヌレート:15          | 102 | A0       |
|        | 19   | 100           | 合成例5:15          | メラミン・シアスレート)15          | 102 | V0       |
|        | 20   | 100           | 合政例6:15          | メラミン・シアヌレート:15          | 103 | Y-0-     |
|        | 21   | 100           | 合成例7:15          | メラミン・シアスレート 115         | 101 | ¥-0      |
| 施      | 22   | 100           | 合成例8:15          | メラミン・シアスレート:15          | 101 | V0       |
|        | 23   | 100           | 合成例9:15          | メラミン・シアヌレート:15          | 102 | V-0      |
|        | 24   | 100           | 合成例 10:15        | メラミン・シアヌレート:15          | 102 | V0       |
|        | 25   | 100           | 合成例 11:15        | メラミン・シアヌレート(15          | 102 | V-0      |
| 91     | 26   | 100           | 合成例 12:15        | スラミン・シアヌレート:15          | 101 | V0       |
|        | 27   | 100           | 合成例3:15          | メラミン・シアスレート 1 15        | 101 | V-0      |
|        | -    |               |                  | ティスモN:15                | ļ   |          |
|        | 28   | 100           | 合波例4:10          | PX-200:2                | 100 | V-0      |
|        | L    |               |                  | メラミン・シアヌレート:15          | ļ   | <u> </u> |
| h:     | 4    | 100           |                  | TPP: 15                 | 92  | V-2      |
|        |      |               |                  | メラミン・シアヌレート: 15         | ļ   | <b></b>  |
| 較      | 2000 | 100           | -                | PX-200:15               | 97  | V-2      |
| 200000 | *    | ļ             |                  | メラミン・シアズレート:15          | 4   |          |
| M      |      | 100           |                  | メラミン・シアヌレート:20          | +   | NG       |
|        | 1    | 100           |                  |                         | 105 | NC       |

[0153] A resinous principle is the mixture of PPE/PS resin =50/50 among a table 2. [0154] Work-example 29 Pori (it is a flame retarder to the resinous principle 100 weight section which consists of 2, the 6-dimethyl 1, 20 copies of 4-phenylene ether, 40 copies of high impact polystyrene (HIPS), and 40 copies of polystyrene (GPPS)) Bridge formation phenoxy phosphazene compound manufactured in the synthetic example 1 It is a melamine SHIANU rate as 20 copies and a fire-resistant assistant. 20 copies are added and they are after mixing and 25mm with a mixer. Fusion kneading was carried out using the biaxial kneading machine, and the flame-retardant-resin constituent was obtained.

[0155] The 1/16-inch-thick test piece was produced for this constituent with injection molding, and heat deflection temperature was measured according to D-648 of the fire-resistant assessment based on the method of examining UL-94, and ASTM. As a result, fire retardancy is V-0, heat deflection temperature is 82 degrees C, and JUSHINGU was not accepted at the time of molding.

[0156] It replaced with the bridge formation phenoxy phosphazene compound manufactured in the work example 30 - the example 1 of 40 composition, and the flame-retardant-resin constituent was obtained like the work example 29 except using the bridge formation phenoxy phosphazene compound manufactured in the synthetic examples 2-12.

[0157] Replace with the bridge formation phenoxy phosphazene compound manufactured in the example 1 of work-example 41 composition, use 20 copies of bridge formation phenoxy phosphazene compounds manufactured in the synthetic example 5, and make it be the same as that of a work example 29 except adding 15 copies of potassium titanate fibers (TISMO-D N) further. The flame-retardant-resin constituent was obtained.

[0158] It replaced with the bridge formation phenoxy phosphazene compound manufactured in the example 1 of work-example 42 composition, and the flame-retardant-resin constituent was obtained like the work example 29 12 copies of bridge formation phenoxy phosphazene

compounds manufactured in the synthetic example 6, and except using four copies of triphenyl phosphate (TPP).

[0159] It replaces with the bridge formation phenoxy phosphazene compound manufactured in the comparative example 8 - the example 1 of 11 composition. TPP 20 copies (comparative example 8) or PX-200 blending 20 copies (comparative example 9), or not blending only a flame retarder — and the loadings of a fire-resistant assistant — 30 copies — carrying out (comparative example 10) — or make it be the same as that of a work example 29 except not blending both a flame retarder and a fire-resistant assistant (comparative example 11) The flame-retardant-resin constituent was obtained.

[0160] Production and assessment of the test piece were performed like the work example 29 using the flame-retardant-resin constituent obtained by the flame-retardant-resin constituent and comparative examples 8-11 which are acquired in the above-mentioned work examples 29-42. These results are shown in a table 3.

[0161] In addition, JUSHINGU with a resin composition object of a comparative example 8 intense at the time of molding was accepted. Moreover, the resin composition object of the comparative example 11 did not show fire retardancy at all.
[0162]

Table 31

| £      |       | <u> </u> |            |                | ومستنسبي | ,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,, |
|--------|-------|----------|------------|----------------|----------|---|
| -      | 00000 | 樹脂成分     | 架橋ホスファ     | 他の雑燃剤、鍵燃助剤     | 熱変形      |   |
|        |       |          | ゼン化合物      | 又は無機質光填剤       | 湿度       |   |
| (3卷)   |       | (4倍)     | (35)       | (部)            | ('C)     | UL-94                                   |
|        | 29    | 100      | 合成例 1 : 20 | メラミン・シアヌレート:20 | 82       | V~0                                     |
|        | 30    | 3(%)     | 合成例2:20    | メラミン・シアヌレート:20 | 81       | V0                                      |
| COMMEN | 31    | 100      | 合成例3:20    | メラミン・シアヌレート:20 | 81       | V-0                                     |
| 窦      | 32    | 100      | 合成例4:20    | メラミン・シアヌレート:25 | 82       | V-0                                     |
|        | 33    | 100      | 合成例 5 : 20 | メラミン・シアヌレート:20 | 82       | V0                                      |
|        | 34    | 100      | 合成例 6:20   | メラミン・シアヌレート:20 | 82       | V-0                                     |
|        | 35    | 100      | 合成例 7:20   | メラミン・シアヌレート:20 | 82       | V0                                      |
| 施      | 36    | 100      | 合成例8:20    | メラミン・シアヌレート:20 | 81       | V-0                                     |
|        | 37    | 100      | 合成例9:20    | メラミン・シアヌレート:20 | 82       | V-0                                     |
|        | 38    | 100      | 合成例10:20   | メラミン・シアスレート:20 | 82       | V-0                                     |
|        | 39    | 100      | 合成例 11:20  | メラミン・シアスレート:20 | 82       | V-0                                     |
| 例      | 40    | 100      | 合政例 12:20  | メラミン・シアスレート:20 | 81       | V-0                                     |
|        | 41    | 100      | 合成例 5 : 20 | メラミン・シアヌレート:20 | 81       | V-0                                     |
|        |       |          |            | ティスモN:15       |          |   |
|        | 42    | 100      | 合成例 6 : 12 | TPF:4          | 80       | V0                                      |
|        |       |          |            | メラミン・シアヌレート:20 |          |   |
| 出      | Š     | 100      |            | TPP : 20       | 75       | V-2                                     |
|        |       |          |            | メラミン・シアヌレート:20 |          |   |
| 較      | 9     | 100      |            | PX-200:20      | 78       | ¥-2                                     |
| *      |       |          |            | メラミン・シアヌレート:20 |          |   |
| 199    | 10    | 100      |            | メラミン・シアヌレート:30 | 86       | NG                                      |
|        | 11    | 100      |            |                | 85       | NG                                      |

[0163] A resinous principle is the mixture of PPE/PS resin =20/80 among a table 3.